

Microwave Oven Method for Rapid Determination of Moisture in Meat

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A new, rapid procedure was developed for moisture determination in meat. Results with the method were evaluated by comparative analysis with AOAC method 24.003(b). The new method is accurate, precise, and simple. Samples were prepared for drying by admixture with ferrous oxide and sodium chloride in glass weighing bottles and heating 2.5 min in a domestic-type 1000 watt microwave oven. After heating, the residues were exposed 1 min in the stream of the oven chamber air blower, then covered and weighed. From comparative determinations on 67 meat samples containing from 3.5 to 77.9% moisture, meat type and moisture level were not significant ($P = 0.05$) sources of variation as determined by *t*-tests. Mean moisture content was 0.05% higher by the microwave oven method than by the AOAC method. Repeatability between duplicates was $\pm 0.47\%$ moisture by microwave oven and $\pm 0.45\%$ by the AOAC method. Precision between paired determinations by the 2 methods was $\pm 0.57\%$ moisture. Both the *t*-test for significance ($P = 0.05$) and linear regression analysis of the comparative determinations indicated that the 2 methods were equivalent for determining moisture. Continued study of the method is recommended.

The need for rapid determination of moisture in the quality control and compliance testing of meat and meat products is of continuing importance to all segments of the meat industry. Rapid methods of analysis should be reasonably accurate, simple, and inexpensive enough for on-line testing by even the small processor. Pettinati *et al.* (1, 2) reviewed available methods for moisture determination more rapid than the official AOAC methods (3). Although some of the data were limited and unconfirmed, the methods were evaluated in the review on the basis of time required, sample size, apparent accuracy and precision, and approximate cost of equipment and operations. The need was discussed for thorough evaluation of many of the methods cited to identify the most effective for use as standards.

In 12 of the cited methods (1, 2), samples of meat and meat products were dried by heating in a conventional oven, below an infrared lamp, or on a hot plate. Moisture determination by means other than radiant heating, specifically by microwaves which create an elevated temperature within the sample, has a background in research which suggests its potential promise for development as an analytical method. Limited data are available on the use of a microwave oven as a rapid heating device and on the determination of moisture based on the measurement of dielectric absorption of microwaves. Davis (4) patented a process for determining moisture and other constituents of meat in which moisture was vaporized from a 70 g sample in 4 min. However, overheating tended to decompose protein and to cause sample loss by spattering. Results of analysis of 4 beef samples containing 59–68% moisture had a mean that was 0.46% high and a standard deviation of $\pm 0.86\%$ moisture relative to determinations by the AOAC method.

A bench-top microwave oven has been designed for moisture determination; it contains a built-in balance and other controls and is commercially available (Apollo Microwave Products) 5407 E. Terra Cotta Ave., Crystal Lake, IL 60014). It has not, however, been systematically tested for moisture determination in meat. Cotterill and Delaney (5) compared use of a microwave oven for determining the moisture content of egg and egg products with a conventional oven. The mean moisture content of 5 samples dried 13 min was 0.4% moisture high and standard deviation was $\pm 0.7\%$ relative to conventional determinations. Carter *et al.* (6), in determining biological solids, dried wastewater samples in a microwave oven for 12–15 min instead of 1 hr in a conventional oven at 103°C. Hesek and Wilson (7) used both 500 and 700 watt microwave ovens to dry samples of inorganic

chemicals analyzed during in-process control. Samples of 20–50 g wet materials were dried in about 15 min instead of 3–4 hr by conventional means. Moisture content of 50 g spent silica gel was reduced from 20 to 1.5% by 30 min of microwave treatment. Organic chemicals were not dried as readily as the inorganics and tended to sublime. The authors advised that overheating could decompose samples and damage the microwave generator.

Studies of the interaction of microwave radiation with the moisture in many agricultural and food materials and measurement of the energy absorbed by the dielectric from an electromagnetic field have led to use of microwaves in many commercial processes, including thawing frozen products, drying, and estimation of moisture content. Applications were reviewed by Nelson (8) who reported that the primary considerations are radiation frequency, which determines the penetration of the radiation, and the temperature dependence of the dielectric properties of the food material. Other variables include nonuniformity of the material; surface and volume geometry, because charges tend to accumulate on sharp projections from the surface; polarization, which can occur at interfacial boundaries; dependence on whether fibers, as in meat, are parallel or perpendicular to the electric field; the effect of bound water on the electrical properties due to the reduced ability of the water molecules to orient freely with an applied electric field; and ash or salt content.

Goldblith (9), using a microwave frequency of 2450 Hz, determined that the dielectric constant (ϵ') of a ground beef sample at 25°C is 52.1 and the dielectric loss factor (ϵ'') is 17.2. For water, the 2 respective values are 78 and 9.8. The dielectric loss of a material expresses how much microwave radiation the material absorbs; it is the sum of the dipole and ionic losses. Because of its ash content, the energy uptake of beef, which resulted in heating, was nearly twice that of water.

Pace *et al.* (10) measured the dielectric properties of 11 commercial fats and oils, including lard, tallow, and bacon fat, and found that the lipid materials absorbed very little microwave radiation. Their ϵ' and ϵ'' values were typically 2.5 and 0.9, respectively. Ohlsson *et al.* (11) studied the relationship of ϵ' and ϵ'' to levels of moisture, fat, and protein in meat as a possible

rapid method for their determination. Measurements of either ϵ' alone or both ϵ' and ϵ'' , with microwaves of either 900 or 2800 MHz, were linearly related ($r = 0.990$) to moisture content. However, the sensitivity of the method was low because 0.5% moisture content corresponded to a difference of 1–2% in ϵ' .

In our investigations, using a domestic-type 1000 watt microwave oven, we soon found that microwave energy applied to meat samples was converted dielectrically to thermal energy so rapidly that the samples spattered. Dispersion of the sample with sand eliminated spattering. However, samples required ≥ 15 min to dry because, as moisture was evaporated, they became more transparent to microwave radiation and traces of moisture evaporated very slowly. Addition of ferrous oxide, a known strong absorber of microwaves, to the dispersing sand resulted in accelerated drying. Drying was further accelerated by substituting granulated sodium chloride for sand. Apparently the sodium chloride caused a dehydration or salting-out effect which promoted water release. In the procedure that was developed and evaluated, 5 g samples of meat were dispersed with a mixture of sodium chloride and ferrous oxide and were dried in a microwave oven by 2.5 min of heating followed by 1 min of air drying. These 2 time periods were selected as optimum by experimentation using comparative moisture determinations by the AOAC method for guidance in their selection. Both heating and air drying times were varied by 15 sec intervals until moisture determinations compared favorably with pre-determined reference values. Once the time periods were established, the comparative analyses reported here were performed. The use of a microwave oven of other design or wattage for moisture determination may require adjustment of the time periods given here.

Microwave Oven Drying

Reagents and Apparatus

(a) *Ferrous oxide*.—Black, powdered (Fisher Scientific Co., 191 S. Gulph Rd., King of Prussia, PA 19406, No. I119).

(b) *Weighing bottle*.—Cylindrical, low form,

Reference to brand or firm name does not constitute endorsement by the U.S. Department of Agriculture over others of a similar nature not mentioned.

with cap-style stopper, 70 mm id \times 33 mm high (Kimble No. 15166, available through laboratory equipment suppliers).

(c) *Glass rod*.—5 mm diameter, 6.5 cm overall length, one end flattened to ca 1 cm diameter (made from glass rod stocked in laboratory).

(d) *Microwave oven*.—Cooking chamber 12" wide \times 12½" long \times 5¼" high, 1000 watt radio-frequency microwave energy output, 2450 MHz (Litton Industries, Minneapolis, MN 55411, Model 500).

Determination

Obtain tare weight, to nearest 1 mg, of weighing bottle, with cap, containing mixture of ca 22 g (heaping teaspoon) granulated NaCl and ca 1.9 g (level teaspoon) ferrous oxide and mashing rod; for pork fatty tissues only, use additional 44 g (2 heaping teaspoons) NaCl to prevent spattering. Into this bottle weigh ca 5 g meat, to nearest 1 mg, ground and mixed according to 24.001. Use mashing rod to thoroughly mix and disperse sample with NaCl-ferrous oxide mixture. (Note: The mixture will assume wetted appearance during thorough mixing as NaCl acts on sample.) Ferrous oxide serves as auxiliary heat generating agent under conditions of microwave irradiation and must be uniformly dispersed to avoid hot spots which may char during heating. Let mashing rod remain in mixture, inclined so that cap can be placed later. Place weighing bottle(s) without cap(s) onto center or near center of oven shelf. Place beaker containing 300–400 ml cracked ice or cold water, covered with plastic wrap and secured with rubber band, on one side of oven shelf and adjacent to oven door.

(Note: This serves as a safety absorber to prevent radiation overload due to small size of sample. Radiation overload will burn out power generator unless oven is equipped with unidirectional shield.) Set oven to heat for 2.5 min. Open oven door after 2.5 min and leave sample in air stream of chamber air blower for 1 min. Lightly place cap on weighing bottle to prevent vacuum lock and weigh. Calculate moisture content from weight loss as percentage of original weight of sample. Disregard irregular white appearance on surface of dried residue, which is powdered salt that dissolved in moisture of sample prior to heating.

Results and Discussion

Moisture determinations on 48 beef and 19 pork samples are shown in Tables 1 and 2. Samples were prepared by mixing lean and fatty tissues. Moisture contents were 3.5–77.9% for beef and 16.6–73.9% for pork. A few samples of individual muscles and fatty tissues were ground and analyzed as such. For example, the first 3 comparative determinations listed in Table 1 were for samples of beef fatty tissue without added lean and the first of the duplicate determinations listed in Table 2 was for a sample of pork fatty tissue. Preliminary experiments established that the use of approximately 22 g sodium chloride and 1.9 g ferrous oxide permitted drying 5 g sample in 2.5 min, an acceptably rapid drying time. Moisture values were significantly higher when samples were reheated for additional 2.5 min periods. For example, samples heated for 3 consecutive 2.5 min drying periods

Table 1. Comparative determinations for per cent moisture in beef by 2.5 min drying in a microwave oven and by AOAC method

Microwave oven ^a	AOAC ^b	Microwave oven ^a	AOAC ^b	Microwave oven ^a	AOAC ^b
3.78	3.50, 3.50	63.06	61.90, 61.92	69.59	69.72 ^a , —
7.28	7.50, 7.60	62.64	62.49, 62.97	68.70	70.00, 70.10
17.44	16.40, 17.10	61.89	63.00, 63.00	69.88	70.27, 69.88
42.04	42.17, 43.09	64.53	63.57, 63.91	69.87	70.01, 70.28
45.03	45.07, 46.18	63.80	63.96, 63.71	70.23	70.57, 69.80
50.09	49.65, 50.01	65.37	64.47, 64.29	70.09	70.34, 70.69
50.37	50.55, 50.15	64.83	64.22, 65.11	70.62	70.67, 70.78
50.91	52.06, 50.75	64.07	65.43, 64.58	70.64	70.89, 70.83
52.87	52.30, 52.03	65.87	65.96, 64.87	71.33	71.19, 70.97
53.34	54.20, 52.80	66.25	65.38, 66.61	71.10	71.60, 71.48
59.32	57.72, 59.66	65.85	66.25, 65.96	72.76	72.10, 71.90
59.86	58.94, 59.04	66.71	66.68, 66.50	72.14	72.52, 71.99
59.02	59.66, 59.71	66.11	66.61, 66.74	75.90	76.31, 75.97
59.88	59.64, 60.05	66.89	66.99, 66.41	76.93	76.59, 76.46
60.41	59.71, 60.01	67.82	67.13, 68.04	76.98	77.66, 77.15
60.71	61.13, 62.17	68.77	69.27, 68.94	77.85	77.96, 77.77

^aSingle determinations.

^bDuplicate determinations.

Table 2. Comparative determinations for per cent moisture in pork by 2.5 min drying in a microwave oven and by AOAC method

Microwave oven ^a	AOAC ^b	Microwave oven ^b	AOAC ^b
59.75	59.80, 60.00	16.21, 16.50	16.81, 16.26
61.94	62.66, 61.79	33.46, 33.01	33.11, 33.23
63.15	62.14, 63.10	40.02, 39.84	39.37, 39.09
66.29	65.00, 65.30	41.96, 41.17	41.86, 40.80
66.03	65.60, 65.10	42.81, 43.09	42.43, 42.32
66.91	66.29, 67.58	43.01, 44.14	43.60, 43.87
66.22	67.35, 67.75	44.78, 45.06	45.07, 45.37
70.68	70.40, 71.34	47.28, 48.29	47.51, 47.86
73.60	73.52, 73.58	51.78, 52.54	51.96, 52.16
73.65	73.61, 74.08		

^aSingle determinations.

^bDuplicate determinations.

averaged 0.05, 0.27, and 0.35% moisture, respectively, higher than AOAC method means. It was shown by *t*-tests ($P = 0.05$) that the difference between means was not significant after the first drying period but was significant after the second and third drying periods. It was concluded that determinations after the first drying were equivalent to those by AOAC method and that reheated samples had been dried beyond optimum values.

To determine whether experimental variables were significant sources of error, an error analysis was performed following the procedures suggested by Youden (12). Repeatability of duplicate determinations (12, p. 17) was determined from the comparative determinations and the data were treated by either paired variate analysis (12, p. 28) or linear regression (12, p. 40) as follows: (1) as a function of kind of meat,

beef or pork, (2) as a function of moisture level, and (3) to determine correlation with AOAC method determinations. All standard deviations that were calculated are reported as \pm per cent moisture and each is an estimate of the 1σ variability of the particular data group.

Results of statistical treatment for kind of meat are summarized in Table 3. The standard deviation between duplicate determinations by the microwave oven method, estimated from data for the 9 samples which were analyzed in replicate (Table 2), was $\pm 0.47\%$ moisture and compared favorably with a value of $\pm 0.45\%$ for duplicate analyses of all samples by the AOAC method. Mean difference between paired results indicated that the accuracy of the microwave oven procedure was equivalent to that of the AOAC method. Specifically, the mean of microwave oven method determinations of moisture was 0.02% low for beef samples, 0.16% high for pork samples, and 0.05% high overall. From the differences between paired results by the 2 methods, the precision relative to AOAC method determination was calculated to be $\pm 0.56\%$ moisture for beef, $\pm 0.58\%$ for pork, and $\pm 0.57\%$ overall. The coefficient of variation, standard deviation as a percentage of the mean moisture content of the samples, indicated that moisture determinations by the 2 methods agreed with a relative precision of $\pm 1\%$ of mean (59.1%) moisture content. The mean differences of paired results, relative to standard error between the 2 methods, were not significant (*t*-tests) at the 95% probability level; the compared methods were not different in determining

Table 3. Statistical analysis of moisture determinations on beef and pork sample groups and overall by 2.5 min drying in a microwave oven and by AOAC method

Type of sample	No. samples	No. detns	Moisture, %							
			Mean		Std dev. between duplicate detns		Results between methods, microwave - AOAC		Microwave vs. AOAC results	
			Micro-wave	AOAC	Micro-wave	AOAC	Mean difference	Std dev.	CV, % ^a	<i>t</i> -value ^b
Beef	48	48	61.07	61.09	—	0.45 ^c	-0.02	0.56	0.91	0.26
Pork	19	28	54.29	54.13	0.47 ^d	0.42	0.16	0.58	1.07	1.44
All	67	76	59.17	59.12	—	0.45 ^e	0.05	0.57	0.96	0.68

^aThe coefficient of variation (CV) here expresses the relative measure of variation between methods and is defined as the ratio of 2 averages, the sample standard deviation and the sample mean: $CV = (100 \text{ std dev.})/\bar{X}$.

^bThese values do not exceed tabular *t*-values at 95% probability level, indicating that the 2 methods determine the same moisture content.

^cDetermined from 47 samples.

^dDetermined from 9 samples.

^eDetermined from 66 samples.

Table 4. Statistics of moisture determinations, grouped by moisture level, of beef and pork samples dried 2.5 min in a microwave oven and by AOAC method

Moisture, %									
Moisture range, %	No. samples	No. detns	Mean		Results between methods, microwave – AOAC			Microwave vs. AOAC results	
					Difference				
			Micro-wave	AOAC	Difference		Std dev.		
					Range	Mean			
3.5–39.11	6	9	23.06	22.84	–0.43–0.91	0.22	0.48	2.12	1.36
41.15–49.83	8	13	44.52	44.40	–0.62–1.06	0.12	0.59	1.33	0.73
50.35–59.90	11	12	55.84	55.69	–0.67–0.87	0.15	0.51	0.91	1.02
61.65–69.72	24	24	65.22	65.19	–1.33–1.15	0.03	0.69	1.06	0.23
70.05–77.86	18	18	72.39	72.54	–1.35–0.76	–0.15	0.43	0.59	1.48

^aThe coefficient of variation (CV) here expresses the *relative* measure of variation between methods. Definition is given in footnote a of Table 3.

^bThese values do not exceed tabular *t*-values at 95% probability level, indicating that there was no significant difference between the values for moisture content obtained by the 2 methods.

moisture of either beef or pork of widely varying moisture contents.

In an additional statistical treatment, the comparative results for both beef and pork samples were grouped into 5 ranges of moisture level to determine accuracy and precision at each level (Table 4). At the lowest moisture level, mean difference between paired results indicated that moisture contents were 0.22% higher by the microwave oven than by the AOAC method. Mean difference tended to decrease as moisture level increased, although at each of the 5 moisture levels the mean difference was substantially less than the respective standard deviation of difference. The latter statistic varied randomly around the overall standard deviation value of $\pm 0.57\%$ moisture (Table 3). The coefficient of variation, 2.12 to 0.59%, tended to decrease as moisture level increased. The *t*-values for comparative data at each moisture level were not significant ($P = 0.05$), indicating that results from the 2 methods were not different at any level.

Linear regression analysis of the entire sets of data (both meats and all levels of moisture) yielded the equation, $Y = 0.995X + 0.29$, in which moisture by microwave (*Y*) is expressed as a function of moisture by the AOAC method (*X*). A high correlation coefficient ($r = 0.9994$) and coefficient of determination ($r^2 = 0.9988$) indicated that 99.88% of the total variation of

moisture by microwave could be attributed to variation of moisture by the AOAC method (covariance) and only 0.12% to random factors. A standard deviation from regression of $\pm 0.56\%$ moisture approximated the standard deviation, calculated by difference analysis, of $\pm 0.57\%$ for all determinations shown in Table 3. The standard error of the intercept, 0.29, was determined to be ± 0.26 and a *t*-test of this value was not significant ($P = 0.05$), which indicated that the intercept did not significantly differ from zero.

Recommendation

The results of this comparative study show that the rapid microwave oven method is as accurate and precise as the AOAC method for the estimation of the moisture content of fresh meat. The Associate Referee is continuing study of the method to evaluate its application to various meat products and recommends that it be studied collaboratively to determine its suitability as an alternative official method.

Acknowledgments

The author thanks his associates for their help, especially Thomas M. Koch and Anthony J. Malloy of this laboratory for technical assistance, and Clifton E. Swift, also of this laboratory, and George Freedman of Raytheon Co., Waltham, MA, for helpful discussions.

This report of the Associate Referee was presented at the 88th Annual Meeting of the AOAC, Oct. 14-17, 1974, at Washington, DC.

The recommendation of the Associate Referee was approved by the General Referee and by Subcommittee C and was accepted by the Association. See (1975) *JAOAC* 58, 315.

REFERENCES

- (1) Pettinati, J. D., Swift, C. E., & Cohen, E. H. (1973) *JAOAC* 56, 544-561
- (2) Pettinati, J. D., Swift, C. E., & Cohen, E. H. (1973) *Proceedings of the 26th Annual Reciprocal Meat Conference*, American Meat Science Association, Pennsylvania State University, University Park, June 17-20, pp. 223-242
- (3) *Official Methods of Analysis* (1975) 12th ed., AOAC, Washington, DC, secs. 24.003(a) and 24.003(b)
- (4) Davis, K. E. (1972) U.S. Patent No. 3,673,852, July 4
- (5) Cotterill, O. J., & Delaney, I. (1959) *Food Technol.* 13, 476
- (6) Carter, J. L., Fleischfresser, D. A., & Ismii, T. K. (1974) *Proceedings of Microwave Power Symposium—1974*, Marquette University, Milwaukee, WI, May 28-31, pp. A3-2/1 to A3-2/4
- (7) Hesek, J. A., & Wilson, R. C. (1974) *Anal. Chem.* 46, 1160
- (8) Nelson, S. O. (1971) Winter Meeting of American Society of Agricultural Engineers, Sherman House, Chicago, IL, *Paper No. 71-847*, Dec. 7-10
- (9) Goldblith, S. A. (1974) *Microwave Energy Appl. Newslett.* 7(3), 9-14
- (10) Pace, W. E., Westphal, W. B., & Goldblith, S. A. (1968) *J. Food Sci.* 33, 30-36
- (11) Ohlsson, T., Henriques, M., & Bengtsson, N. E. (1974) *J. Food Sci.* 39, 1153-1156
- (12) Youden, W. J. (1951) *Statistical Methods for Chemists*, John Wiley & Sons, New York